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IS 11720-13 (2001): Methods of Test for Synthetic Rubber, Part 13: Determination of Gel Content [PCD 13: Rubber and Rubber Products]



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“Knowledge is such a treasure which cannot be stolen”



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भारतीय मानक  
संश्लेषित रबड़ की परीक्षा विधियाँ  
भाग 13 जेल अंश ज्ञात करना

*Indian Standard*

**METHODS OF TEST FOR  
SYNTHETIC RUBBER**

**PART 13 DETERMINATION OF GEL CONTENT**

ICS 71.040.40 : 83.060

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**BUREAU OF INDIAN STANDARDS**  
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NEW DELHI 110002

## FOREWORD

This Indian Standard (Part 13) was adopted by the Bureau of Indian Standards, after the draft was finalized by the Rubber Sectional Committee and had been approved by the Petroleum, Coal and Related Products Division Council.

The concerned committee has decided to prepare common methods of test for synthetic rubber under SR (Synthetic Rubber) series, namely, IS 11720 and this will be applicable to all types of synthetic rubbers being produced indigenously. This standard (Part 13) is the thirteenth in the series. The other standards of this series are as follows:

- Part 1 Methods of test for synthetic rubber: Part 1 Determination of antioxidants (SR:1)
- Part 2 Methods of test for synthetic rubber: Part 2 Measurement of vulcanization characteristics with oscillating disc curemeter (SR:2)
- Part 3 Methods of test for synthetic rubber: Part 3 Determination of mooney viscosity
- Part 4 Methods of test for synthetic rubber: Part 4 Determination of volatile matter
- Part 5 Methods of test for synthetic rubber: Part 5 Determination of ash
- Part 6 Methods of test for synthetic rubber: Part 6 Determination of solvent extract
- Part 11 Methods of test for synthetic rubber: Part 11 Rubber raw styrene-butadiene — Determination of soap and organic-acid content

In the preparation of this standard, considerable assistance has been derived from ASTM D 3616.

The Composition of the Committee responsible for formulation of this standard is given in Annex A.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'

*Indian Standard***METHODS OF TEST FOR  
SYNTHETIC RUBBER****PART 13 DETERMINATION OF GEL CONTENT****1 SCOPE**

**1.1** This test method covers the determination of the gel characteristics of raw non-oil-extended and non-pigmented SBR and NBR.

**1.2** This test method may be used to determine the gel characteristics of rubbers other than SBR and NBR, however, solvents other than 2-butanone and toluene may be required.

**1.3** This test method is not intended for the measurement of micro-gel.

**2 QUALITY OF REAGENTS**

Unless specified otherwise, pure chemicals shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

**3 APPARATUS**

**3.1** Borosilicate weighing bottle, 45/12 standard taper, 40 mm in inside diameter, 100 mm high, with cover.

**3.2** Screen rack, consisting of five 300-micrometer (No. 50) circular screens mounted on a stainless steel tube to fit the weighing bottle (3.1). Borosilicate bottle, cover, and rack will be referred to hereafter as 'that unit'. (Fig. 1).

**3.3** Pipette, capable of fitting within the tube of the screen rack 3.2.

**3.4 Disposable Aluminium Dishes**

**3.5** Balance, capable of accurately weighing to  $\pm 0.1$  mg.

**3.6** Screw-cap bottles, 100 ml minimum capacity, or erlenmeyer flasks, 125 ml capacity.

**3.7 Borosilicate Wool of Cotton**

**3.8** Additional glassware, sufficient to carry out the procedure as written. Glass 'A' pipettes of 25 and 100 ml volume are mandatory.

**4 REAGENTS**

**4.1** 2-Butanone (methyl ethyl ketone), for NBR rubbers,

**4.2** Toluene, for SBR Rubbers

**5 SAMPLING**

**5.1** Select a representative sample of the rubber to be tested.

**5.2** Do not mill the sample prior to testing except for rubbers in the powdered form (*see* 6.2).

NOTE — The presence of dusting agents may result in erroneous values for total solids.

**6 PROCEDURE**

**6.1** Using a clean scissors, cut the sample into strips less than 1 mm in thickness and about 5 mm long.

**6.2** In the case of rubbers in powdered form, the sample may be massed or compacted by the use of a hand press with platens at 50°C. It may then be cut into appropriate strips. Rubbers that will not fall through the screen may be placed on the screen without prior treatment.

**6.3** Weigh 0.39 to 0.41 g of the prepared sample to the nearest 0.1 mg and distribute evenly over the four lowest screens of the unit. Place the pieces near the centre of each screen.

**6.4** Gently place the screen rack into the borosilicate weighing bottle and deliver 100 ml of the chosen solvent into the bottle. Tip the container to assure complete wetting of each screen.

**6.5** Cap the bottle and allow to stand for 16 to 20 h at  $25 \pm 2^\circ\text{C}$ , in the dark.

**6.6** After 16 to 20 h, remove the present cap from the unit, lift the screen and examine for visible gel.

NOTE — Some rubbers will not go into solution as readily as others. It is necessary for the analyst to distinguish between undissolved polymer and true gel. A thinner or more porous sample will aid the solution and an extending standard period up to 48 h may be helpful in distinguishing gel from true polymer solution.

**6.7** Pipette the liquid from the unit into a screw cap bottle or erlenmeyer flask by inserting the pipette through the centre of the screen rack. Filter the liquid through cotton or borosilicate glass wool.

**6.8** Pipette exactly 25 ml of this liquid into a dried and weighed aluminium dish and place the dish on a steam or electric hot plate at  $100 \pm 10^\circ\text{C}$ . Alternately heat and weigh the dish to the nearest 0.1 mg, until a constant mass is achieved. Record this value.

**7 CALCULATION**

Calculate gel content as follows:

$$m_1 \times 4 = m_2$$

$$\text{Gel, percent by mass} = \frac{(m_3 - m_2)}{m_3} \times 100$$

where

$m_1$  = Mass of the dried solution, 25 cm<sup>3</sup> volume,

$m_2$  = Mass of the total dried solution, and

$m_3$  = Mass of the original sample.

**7.1** A test result is the average of two determinations.

## ANNEX A

( Foreword )

## COMMITTEE COMPOSITION

## Rubber Sectional Committee, PCD 14

<i>Organization</i>	<i>Representative(s)</i>
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(Continued on page 4)



# IS 11720 (Part 13) : 2001

(Continued from page 3)

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